PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Phosphatide Anti-spattering Agents for Margarine

We, UNILEVER LIMITED, a Company registered under the law of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement: —

This invention relates to a process for preparing phosphatide anti-spattering agents and to margarine containing them.

Margarine is commonly used for frying. For this purpose it is desirable that margarine shall spatter vary little and preferably not at all that it shall give a fine sediment which does not adhere to the frying pan, and that it shall have an attractive golden-yellow colour after frying. These properties are not normally imparted by any of the usual constituents of margarine, such as fats and the mono- and di- glycerides normally employed as emulsifiers.

In margarine the aqueous phase may or may not be derived from milk. A milk margarine contains proteins derived from the milk, which improves the frying properties of the margarine in one respect in that during frying they play a part in the formation of a brown sediment which is much valued. This sediment is, however, normally not finely dispersed. The ability of margarine to lose its water during frying without spattering can to some degree be imparted to a water margarine by incorporating commercial lecithin. The addition of lecithin to a milk margarine, on the other hand, does not greatly reduce its tendency to spatter, apparently owing to some interaction between the lecithin and the milk protein. Reduction of the tendency to spatter of protein-containing margarine, especially such a margarine that is substantially free from air and salt, has been a problem to which hitherto no altogether satisfactory solution has been found.

The present invention is concerned with reducing the tendency to spatter of a margarine, especially a milk protein-containing margarine, and one that is substantially free from air and salt. The invention provides a method for achieving this by incorporating in the margarine certain emulsifiers present in the crude phosphatides of vegetable oils.

Vegetable phosphatides are highly complicated compositions comprising a large number of compounds. Owing to this complicated structure it is very difficult to obtain from crude phosphatides fractions having closely defined properties, because a very slight modification in the fractionation process may lead to very pronounced differences in the effectiveness of the final product, as compounds which are present only in very small amounts may interact with other compounds. In crude soyabean phosphatides the main constituents are phosphatidylcholine (often by itself referred to as lecithin), phosphatidylethanolamine (often referred to as cephalin), phosphoinositide (often referred to as lipositol) and phosphatidic acids. Other phosphatides, such as phosphatidylserine and sphingomyelin are also present together with carrier soyabean oil and soyabean fatty acids, sterols, tocopherols, and free carbohydrates. Of these compounds phosphatidyl choline, soyabean oil fatty acids, sterols and tocopherols are easily soluble in ethanol, while phosphatidylethanolamine and soyabean oil are far less soluble in ethanol, so that under particular conditions different products can be obtained by means of extraction with ethanol, particularly as the various com-

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pounds have an effect on each other's solubility. As such particular conditions can be mentioned the temperature during extraction and separation, the kind and the concentration of the solvent used, the amount of phosphatide in relation to the solvent, and the influence of various additives before or after the extraction.

It has been proposed in U.S. Patent 2,640,780 to incorporate into margarine an anti-spattering agent obtained from vegetable phosphatides by a process in which the phosphatides are extracted with sufficient of an alcohol containing up to 4 carbon atoms in the molecule to extract all alcohol-soluble materials, carrier oil being excluded from the phosphatides. The removal of the carrier oil is said to be an essential step to remove along with it a substantial amount of deleterious impurities affecting the activity of the final product.

It has now been found that under particular extraction conditions a product with better anti-spattering properties, especially for milk margarine, than those of the product of U.S. Patent 2,640,780 can be obtained if the carrier oil in the crude phosphatide is not removed.

In a process of the invention a crude undefatted vegetable oil phosphatide (that is, a crude phosphatide containing its carrier oil) is extracted with an 80 to 95% aqueous alkanol of from 1 to 3 carbon atoms in the molecule in an amount of from 2 to 10 litres per kilogram of crude phosphatide at a temperature within the range of from 10 to 30°C, and the aqueous alcohol is removed from the extracted material to give a phosphatide in which the proportion of phosphatidylcholine to phosphatidylethanolamine is between 4 and 5.5 to 1 by weight.

The resulting phosphatide is very effective in preventing spattering and the addition of only very small amounts of it to milk protein-containing margarine is required. On frying with the resulting margarine a very fine sediment and a stable foam resembling that of fried butter is obtained. It appears likely that the advantages found are connected with the retention in the phosphatide of sterols, which would be removed with the carrier oil by the process of U.S. Patent 2,640,780.

Preferably the alkanol used is ethanol, and preferably the crude vegetable phosphatide is a soyabean oil phosphatide, but other phosphatides are also suitable, for example, rapeseed oil phosphatide.

In a batch process using ethanol 3 litres per kilogram of crude phosphatide gives particularly good results, while in a counter-current continuous process 7 litres ethanol per kilogram may be the optimum. Preferably from 1 to 5% by weight of soyabean oil fatty acid is added to the crude vegetable phosphatide before the extraction in order to reduce the viscosity of the phosphatide: the solubility of the alcohol-soluble fraction in margarine oils is also substantially improved by the fatty acids thus incorporated in the extract.

In carrying out the process the aqueous alcohol and the crude vegetable phosphatide are stirred together intensively. To obtain equilibration of distribution, it is necessary to mix them thoroughly for some time, ½—2 hours being usually sufficient. The mixture is allowed to stand to form two phases, which are carefully separated. It is important to separate the phases carefully, because small amounts of less soluble components from the non-alcoholic phase, particularly additional phosphatidylethanolamine, considerably reduce the effectiveness of the product. The alcohol is then conveniently eliminated under reduced pressure at 30 to 60°C. In order to carry out the process economically, it has been found that the use of about 90% ethanol is convenient as the ethanol can be recovered in almost the initial concentration very easily by distillation, so that it can be re-used immediately with only slight adjustment in the concentration by adding either 96% aqueous ethanol or water.

Preferably the extraction is performed in one step. The proportion of the phosphatide obtained is preferably from 15 to 25% by weight of the crude vegetable phosphatide, and the conditions are adjusted to achieve this where required.

The invention includes a margarine, preferably one which is substantially salt-free and air-free, and especially one whose aqueous phase contains milk protein, which contains from 0.02 to 0.6%, preferably 0.04 to 0.1%, by weight of an anti-spattering agent obtained by a process of the invention, the amount of the phosphatidylcholine present being at least 0.01% by weight of the margarine. The anti-spattering agent can be incorporated either in the fat phase or in the aqueous phase of the margarine. About 0.05% of milk protein by weight of the margarine (equal to about 2% by weight of milk) is preferably present in the margarine.

The following Examples illustrate the invention. The amounts of phosphatidylcholine and phosphatidylethanolamine were determined by the method described by H. Wagner in *Fette, Seifen, Nahrungsmittel*, 1961, *62*, 1119. Proportions of phosphatidylcholine to phosphatidylethanolamine by weight are expressed as PC : PE.

The frying properties of the margarines obtained according to the Examples were tested by the following procedure. A pan of diameter 18 cm. was preheated to 200°C. A sheet of filter-paper was fixed horizontally 20 cm. above the bottom of the pan, and 50 g. of margarine were put into the pan

and heated until the evaporation of water had ceased. Spattered fat was caught by the filter-paper, forming more or less coherent fat spots on it. The extent of these fat spots is a measure of the spattering.

EXAMPLE 1

1 Kilogram of crude undefatted soyabean phosphatide was thoroughly mixed at 20°C. with 3 litres of 90% aqueous ethanol until a homogenous dispersion of the phosphatide was obtained. After providing a resting period of about 2 hours to effect phase separation, the clear alcoholic upper phase was siphoned off and the solvent distilled off from it at 45°C. and at reduced pressure (10 mm Hg). In this way 155 g. (15.5%) of a phosphatide anti-spattering agent was obtained containing 33.6% phosphatidylcholine and 8% phosphatidylethanolamine by weight (PC : PE=4.2 : 1).

A substantially air- and salt- free margarine containing 5% by weight of milk was prepared by conventional methods using 0.08% of the phosphatide anti-spattering agent by weight of the margarine dissolved in the fat phase. In the fresh condition and also after 8 weeks storage at 15°C. the margarine did not spatter on frying and produced a finely divided sediment and a stable foam resembling that of fried butter.

A similar product was produced when the proportion or milk was increased to 15%.

EXAMPLE 2

5 Kilograms of crude undefatted soyabean phosphatide to which 200 g. of a soyabean oil fatty acid had been added was stirred for 2½ hours at 15°C. with 17.5 litres of 91% aqueous ethanol. After a 5 hours' resting period the phases were carefully separated, and the solvent was distilled off from the alcoholic phase. In this way 0.7 kg. was obtained of a phosphatide anti-spattering agent containing 44.1% of phosphatidylcholine and 9% of phosphatidylethanolamine by weight (PC : PE=4.9 : 1).

A substantially air- and salt- free milk margarine containing 0.06% by weight of the phosphatide anti-spattering agent incorporated in the fat phase was prepared: the product did not spatter.

EXAMPLE 3

In a modification of the process described in Example 3, the phosphatide anti-spattering agent was dispersed in the milk-containing aqueous phase. This also resulted in a non-spattering margarine.

EXAMPLE 4

In a countercurrent extraction apparatus consisting of 4 separation units (manufactured by the German firm of Westfalia, type Lg 205) 1.58 kg. crude undefatted soyabean phosphatide at a temperature of 15°C. was extracted with 6 litres 90% aqueous ethanol at 12°C. After distilling off the solvent from the alcohol phase at reduced pressure (4 mm Hg at 40—45°C.), there was obtained 0.27 kg. (17.1%) of a phosphatide anti-spattering agent containing 49.6% phosphatidylcholine and 9.1% phosphatidylethanolamine by weight (PC : PE=5.45 : 1).

Air- and salt- free milk margarines were prepared incorporating 0.04%, 0.06% and 0.08% by weight, respectively, of the phosphatide anti-spattering agent. The behaviour of these margarines on frying was progressively better with increasing amounts of anti-spattering agent: at 0.04% there was only slight spattering and at 0.08% no spattering at all.

EXAMPLE 5

In a Jantzen distribution column as described by S.O. Judermann in Houben-Weyl, Methoden der Organischen Chemie, Volume 1/1, page 223 ff., of 4 to 5 theoretical plates, 600 g. crude undefatted soyabean phosphatide was continuously extracted at 18—20°C. with 3.06 litres 93% aqueous ethanol. After removal of the solvent from the alcohol extracts 134 g. (22.3%) of a phosphatide anti-spattering agent containing 32.6% phosphatidylcholine and 6.4% phosphatidylethanolamine by weight (PC : PE= 5.1 : 1) was obtained.

A non-spattering air- and salt- free milk margarine was prepared incorporating 0.07% by weight of the phosphatide anti- spattering agent.

WHAT WE CLAIM IS:—

1. A process for preparing a phosphatide anti-spattering agent for margarine, in which a crude undefatted vegetable phosphatide is extracted with an 80 to 95% aqueous alkanol of from 1 to 3 carbon atoms in the molecule in an amount of from 2 to 10 litres per kilogram of crude phosphatide at a temperature within the range of from 10 to 30°C, and the aqueous alcohol is removed from the extracted material to give a phosphatide in which the proportion of phosphatidylcholine to phosphatidylethanolamine is between 4 and 5.5 to 1 by weight.

2. A process according to Claim 1, in which the alkanol is ethanol.

3. A process according to Claim 1 or Claim 2, in which the crude vegetable phosphatide is a soyabean oil phosphatide.

4. A process according to Claim 3, in which the crude vegetable phosphatide is one to which from 1 to 5% by weight of soyabean oil fatty acid has been added before the extraction.

5. A process according to any one of Claims 1 to 3, in which the proportion of the phosphatide obtained is from 15 to 25% by weight of the crude vegetable phosphatide.

6. A process according to any preceding claim, in which the extraction is performed in one step.

7. A process according to Claim 1, substantially as described in any one of Examples 1, 2, 4 and 5.

8. A phosphatide anti-spattering agent when prepared by a process according to any preceding claim.

9. A margarine containing from 0.02 to 0.6% by weight of an anti-spattering agent according to Claim 8, the amount of phosphatidylcholine present being at least 0.01% by weight of the margarine.

10. A margarine according to Claim 9 which is substantially salt-free and air-free.

11. A margarine according to Claim 9 or Claim 10 and whose aqueous phase contains milk protein.

12. A margarine containing a phosphatide anti-spattering agent and substantially as described in any one of Examples 1 to 5.

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